

California Environmental Protection Agency



Air Resources Board

SOP MLD 020

**STANDARD OPERATING PROCEDURE FOR
CLEANING 6-LITER SUMMA POLISHED CANISTERS**

Engineering and Laboratory Branch
Monitoring and Laboratory Division

First Approved Date of SOP: March 1, 1997
Approval Date of Last SOP Amendment: October 29, 1999
Revision Number: 3.2

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PROCEDURE FOR CLEANING 6-LITER SUMMA™ POLISHED CANISTERS

1. METHOD INFORMATION

1.1 Scope

This document describes the method for cleaning 6-liter SUMMA™ passivated canisters used for ambient sampling of toxic volatile organic compounds (VOCs), and non-methane hydrocarbons (NMHC). This SOP also describes a method to evacuate and pressurize individual canisters or groups of canisters that do not require cleaning.

1.2 Summary of Method

Eight 6-liter canisters are connected to manifolds in two ovens and evacuated to less than 1.0 torr (1.0 mm Hg). The canisters are heated to 55°C and are then purged five times with humidified grade 5 ultra pure nitrogen (99.9999% pure) that is additionally cleansed using an Aadco Air Purifier. A liquid nitrogen cold trap on the vacuum line prohibits back-diffusion of the vacuum pump oil vapor, and also prevents water vapor from entering the vacuum pump. Both manual and automated procedures are provided. This method is based on EPA Method TO-14A.

Cleaned canisters must meet the criteria levels listed in Table 1 for toxic VOCs and NMHC.

1.3 Interferences/Limitations

- 1.3.1 Periodically, canisters used for standards or controls may need reconditioning.
- 1.3.2 Canisters containing high VOC concentrations that are difficult to clean and may require more than one cleaning session to meet specified contamination criteria.

1.4 Apparatus

- 1.4.1 Aculife treated stainless steel tubing 1/2", 1/4", and 1/8" (Scott Specialty Gases).
- 1.4.2 Duo-Seal, two stage, vacuum pump, Sargent-Welch, Model 1397B-01.
- 1.4.3 Equatherm ovens, Curtin Matheson Scientific, 0-325°C, Model # 213-405 or forced convection ovens, VWR Scientific, 40-240°C, Model 1370FD.

- 1.4.4 Dewar, cylindrical, 1600 ml capacity, 80 mm ID, Kontes KM-611410-2116.
- 1.4.5 Vacuum trap, 25 mm O.D., body length 275 mm, Kontes K-926050-0025.
- 1.4.6 Aadco air purifier, Model 737-42.
- 1.4.7 Gauges, 0-20 torr and 0-2600 torr, Wallace and Tiernan, Models 61C-1D0020 and 61A-1A-0050D.
- 1.4.8 Aluminum sheet, 49" x 40" x 1/4".
- 1.4.9 Valves, 1/2" Nupro, Model #SS-8UW (quantity of 6 for each cleaning system).
- 1.4.10 Valves, 1/4" Nupro, Model #SS-4H (8 for 2 ovens).
- 1.4.11 Fittings, Swagelok.

Description	Quantity
1/2" union cross	2
1/2" union tee	4
1/4" reducing union	7
3/4"-1/2" reducing union	1
1" dia ultra torr union	2
1" dia SS flex hose	2
1" dia ultra torr male connectors	2
1/2" female connectors	2
1/2"- 1/4" reducing adaptor	2

- 1.4.12 Lab Jacks 6" x 6" x 11" Fisher C # 14-673-51.
- 1.4.13 30 g pressure release valve.
- 1.4.14 Safety glasses, cryogenic gloves.
- 1.4.15 Canister conditioner controller (customized to meet ARB specifications), North Star Instruments (See Appendix Part A: Description and Operation, Part B: Major Components, and Part C: Restarting Controller after Power Failure).
- 1.4.16 Solenoid valves (normal open type, quantity of 4 for each canister cleaning system).
- 1.4.17 Mass flow controller, Porter Instrument Company, Inc., MDL. F202MFC or Tylan RO-32.

- 1.4.18 Humidifier Canister. i.e. a 6L SUMMA™ passivated canister filled with ≥ 500 ml of HPLC grade water.

1.5 Materials

- 1.5.1 Compressed grade 5 ultra pure nitrogen that is further cleansed by passing it through an Aadco purifier.
- 1.5.2 Liquid nitrogen.
- 1.5.3 HPLC grade water.
- 1.5.4 Vacuum grease.
- 1.5.5 Vacuum pump oil.

1.6 Precautions

- 1.6.1 Use safety glasses and gloves when handling liquid nitrogen.
- 1.6.2 Do not expose the low pressure gauge to high pressure (greater than 20 torr).
- 1.6.3 Do not pressurize the canisters to more than 30 psig.
- 1.6.4 Do not over tighten the valves on the canisters.
- 1.6.5 Keep the 1600 ml liquid nitrogen dewar filled whenever the vacuum pump is running.
- 1.6.6 Do not allow trapped vacuum vapors to travel into the clean part of the system. Always have the gas travel from the clean part of the system to the dirty part of the system.
- 1.6.7 Check vacuum pump oil level periodically. Change oil every six months.
- 1.6.8 Ensure that the humidification system (system bubbler) always contains greater than 500 ml of water for proper humidification by weighing the humidifier canister annually.
- 1.6.9 All canisters containing standard or control samples must be vented in the safety fume hood before cleaning to avoid contamination of the cleaning system.
- 1.6.10 The nitrogen cylinder should be changed whenever the cylinder pressure drops below 500 psig.
- 1.6.11 The nitrogen cylinder output pressure should not exceed 40 psig.

2. AUTOMATED PROCEDURE TO CLEAN A GROUP OF EIGHT SUMMA POLISHED CANISTERS

2.1 Summary of Automated Procedure to Clean Canisters

Automation of the canister cleaning system has been achieved by using solenoid valves in place of the manually operated valves on the existing system. All pressure and time-based events of the cleaning procedure are operated by a Canister Conditioner Controller (CCC), which allows the cleaning procedure to complete evacuation and purge cycles in the absence of the operator. Toggle switches on the CCC offer a choice of manual override capability for the inlet valves.

SUMMARY OF STANDBY CONDITIONS

Valves: 1, 2, 5, 7	: CLOSED
Valves: 3, 4, 6,	: OPEN (positive flow through V2 orifice)
Oven Manifold Valves	: CLOSED
Liquid N ₂ Valve	: CLOSED
Aadco Purifier	: ON or OFF
Pump	: OFF
Pressure Gauge	: 760 torr (atmospheric pressure)

2.2 Canister Cleaning Procedure

Please refer to Figures 1-1 and 1-2 to interpret all component symbols and structures. Please note that all toggle switches are located on the Canister Conditioner Controller (See Figure 2).

- 2.2.1 Turn the Aadco Air Purifier “**ON**”.
- 2.2.2 Close all solenoids by pushing the **STANDYBY/CYCLE/VENT** toggle (#1) into the middle position (see Figure 2).
- 2.2.3 Open the compressed nitrogen cylinder valve as well as the nitrogen regulator needle valve.
- 2.2.4 Set the mass flow controller (MFC) to 12 for the Tylan RO-32 or to 60% for the Porter Instrument.
- 2.2.5 Place the 1600 ml dewar underneath the glass vacuum cold trap such that the glass finger is inside the dewar but not touching the inner surface of the dewar.
- 2.2.6 Secure all supports for the dewar-trap apparatus.

CAUTION: SAFETY GLASSES & GLOVES ARE REQUIRED FOR STEP 2.2.7

- 2.2.7 **SLOWLY** fill a second dewar with liquid nitrogen, then **CAREFULLY** transfer the liquid nitrogen to the secured dewar. The level of liquid nitrogen should be about 1" from the top of the dewar.
- 2.2.8 Ensure that **V2** (vacuum trap purge valve) is closed.
- 2.2.9 Ensure that **V1** (vacuum pump inlet) is closed, turn on the vacuum pump.
- 2.2.10 **OPEN V1** manually.
- 2.2.11 Vent all eight pre-selected canisters in the safety fume hood before attaching them to the manifolds.
- 2.2.12 Record the canister numbers into the cleaning logbook according to the oven manifold number (labeled in oven).
- 2.2.13 Connect as many as eight 6-liter canisters to the oven manifolds with the canister sampling valves closed. Place canisters in the back of the oven first. Tighten each manifold connection to the canister employing a wrench (size 9/16).
- 2.2.14 **OPEN** canister sampling valves. The pressure of the system will rise slightly.
- 2.2.15 **OPEN** all oven manifold valves.
- 2.2.16 Turn on the ovens, which should be preset at 55⁰C. Close and latch oven doors.
- 2.2.17 Switch **EVAC/AUTO/FILL** toggle (#10) to the left position (**EVAC ON**). This will evacuate the system to the oven manifold to check for system leaks.
- 2.2.18 When the pressure gauge of the canister cleaning system reads <**20 torr**, **OPEN V5** (to vacuum gauge) to ensure that the system is able to obtain an evacuated state of <1.0 torr.
- 2.2.19 If there are no leaks attributed to the canister-oven manifold connections, the system should evacuate and the digital pressure readout on the CCC should read < or = 0.0 mm Hg.
- 2.2.20 **CLOSE V5.**
- 2.2.21 Switch **AUTO/EVAC/FILL** toggle (#10) to the middle position (**AUTO ON**). This will maintain the evacuated state of the system.
- 2.2.22 Set **DRY/HUMID** toggle (#2) to "HUMID".
- 2.2.23 Keep timer A (#3) at 0 minutes and set timer B (#4) to 7 minutes hold time for pressurization.

- 2.2.24 Set Auto Advance toggle (#5) to "ON".
- 2.2.25 Make sure the thumb wheel (#6) is set on "5", which permits the cleaning procedure to complete five full evacuation and humidified nitrogen purge cycles without supervision. Press the **LOAD** toggle (#7).
- 2.2.26 Toggle the **RUN** switch (#8) from "**STOP**" to "**RUN**" to start cleaning. It takes about **one hour and a half** to complete the five full cycles.
- 2.2.27 The cleaning process will be done when the cycle counter (#9) reads "0" and the **EVAC LED's** are lit. To ensure that the canisters are at vacuum, open the oven door and determine if all canister pressure gauges are < -30 " Hg.
- 2.2.28 **CLOSE** canister sampling valves and oven manifold valves, then press **STOP** (# 8) to end program routine. This will close all solenoid valves.
- 2.2.29 If the canister(s) require **PRESSURIZATION**, proceed to the procedures for pressurizing evacuated canisters (Section 2.3). If the canister(s) must remain **EVACUATED**, proceed to the Post Cleaning Procedure (Section 2.6).
- 2.2.30 One canister per cleaning batch must be documented and analyzed by the gas chromatographic method for which they are used. The contamination check results for each batch must not exceed the established criteria listed in Table 1.

2.3 To Pressurize Evacuated Canisters After Cleaning

- 2.3.1 **OPEN ONLY** valves to canisters that require pressurizing or filling.
- 2.3.2 **CLOSE V3** (nitrogen inlet) and **V4** (vacuum inlet). Switch **EVAC/AUTO/FILL** toggle (#10) to the right position (**FILL ON**).
- 2.3.3 **SLOWLY, OPEN V3.**
- 2.3.4 When the desired final pressure has been reached, **CLOSE** the canister sampling valves and oven manifold valves, then **CLOSE V3**.
- 2.3.5 Switch **EVAC/AUTO/FILL** toggle (#10) to the middle position (**AUTO ON**).
- 2.3.6 **OPEN V3** and **V4**.

2.3.7 Proceed to the Post Cleaning Procedure (Section 2.6).

SUMMARY OF STANDBY CONDITIONS

Valves: 1, 2, 5, 7	: CLOSED
Valves 3, 4, 6	: OPEN
Oven Manifold Valves	: CLOSED
Liquid N ₂ Valve	: CLOSED
Aadco Purifier	: ON or OFF
Pump	: OFF
Pressure Gauge	: 760 torr (atmospheric pressure)

2.4 To Evacuate Canisters (From Standby Mode)

2.4.1 Close all solenoids by pushing the **STANDBY/CYCLE/VENT** toggle (#1) into the middle position (See Figure 2).

2.4.2 Place the 1600 ml dewar underneath the glass vacuum cold trap such that the glass finger is inside but not touching the inner surface of dewar.

2.4.3 Secure all supports for the dewar-trap apparatus.

CAUTION: SAFETY GLASSES & GLOVES ARE REQUIRED FOR STEP 2.4.4

2.4.4 **SLOWLY** fill a second dewar with liquid nitrogen, then **CAREFULLY** transfer the liquid nitrogen to the secured dewar. The level of liquid nitrogen should be about 1 inch from the top of the dewar.

2.4.5 Ensure that **V1** (vacuum pump inlet) and **V2** (vacuum trap purge valve) are closed.

2.4.6 Turn on vacuum pump.

2.4.7 **OPEN V1** manually.

2.4.8 Connect as many as eight 6-liter canisters to the oven manifolds with the canister sampling valves closed. Place canisters in the back of the oven first and tighten them down using a wrench (size 9/16").

2.4.9 The ovens do **not** need to be **on** for this procedure.

2.4.10 **OPEN** oven manifold valves.

2.4.11 Switch **Evac/Auto/Fill** toggle (#10) to the left position (**EVAC ON**). This will evacuate the system to the oven manifold to check for system leaks.

- 2.4.12 When the pressure gauge of the cleaning system reads **<20 torr or < 1.0 psig, OPEN V5** (to vacuum gauge) and ensure that the system can evacuate to **<1.0 torr**.
- 2.4.13 **CLOSE V5** and leave the **Evac/Auto/Fill** toggle (#10) in the left position (**EVAC ON**).
- 2.4.14 **OPEN** the canister sampling valves.
- 2.4.15 When the digital pressure gauge of the Canister Conditioner Controller drops below **1 torr**, **CLOSE** the canister sampling valves and oven manifold valves.
- 2.4.16 Switch the **Evac/Auto/Fill** toggle (#10) to the middle position (**AUTO ON**).
- 2.4.17 **CLOSE V3** (nitrogen inlet), and switch **Evac/Auto/Fill** toggle (#10) to the right position (**FILL ON**).
- 2.4.18 **SLOWLY OPEN V3** and pressurize the canister cleaning system to approximately 760-800 torr as indicated by the digital pressure gauge of the Canister Conditioner Controller.
- 2.4.19 **CLOSE V3** and switch **Evac/Auto/Fill** toggle (#10) to the middle position (**AUTO ON**).
- 2.4.20 **OPEN V3**.
- 2.4.21 **CLOSE V1** (vacuum pump valve).

CAUTION: *SAFETY GLASSES & GLOVES ARE REQUIRED FOR STEP 2.4.22*

- 2.4.22 Remove the dewar from the cold trap apparatus and dispose of the liquid nitrogen according to the current safety guidelines.
- 2.4.23 Close the compressed nitrogen cylinder valve as well as the nitrogen regulator needle valve.
- 2.4.24 Remove the canisters from the ovens.
- 2.4.25 Enter the appropriate information (i.e. contamination check number) into the Canister Logbook.
- 2.4.26 Turn the Aadco purifier off only if the cleaning system will not be used for at least two days.

2.5 To Pressurize Canisters (From Standby Mode)

- 2.5.1 Turn the Aadco Air Purifier **"ON"**.
- 2.5.2 Leave the **STANDBY/CYLCE/VENT** toggle (#1) in the middle position to

ensure that all solenoid valves are closed.

- 2.5.3 Set the mass flow controller (MFC) to 12 for the Tylan RO-32 or to 60% for the Porter Instrument.
- 2.5.4 Proceed with Section 2.4 (To Evacuate Canisters), Steps 2.4.03-2.4.13. Procedures 2.4.11-2.4.13 are done to avoid any possible contamination to the canister from the system due to leaks, etc...
- 2.5.5 Set the **DRY/HUMID** toggle (#2) to "HUMID".
- 2.5.6 Pressurize the pre-selected canister(s) by opening the canister sampling valves and oven manifold valves. The system and canister(s) should initially be <1.0 torr.
- 2.5.7 **CLOSE V3** (nitrogen inlet) and **V4** (vacuum inlet).
- 2.5.8 Switch **Evac/Auto/Fill** toggle (#10) to the right position (**FILL ON**).
- 2.5.9 **SLOWLY OPEN V3** and allow the canister(s) to pressurize to the required level indicated on the pressure gauge(s) of the canister(s).
- 2.5.10 **CLOSE** the canister sampling valves and oven manifold valves.
- 2.5.11 **CLOSE V3.**
- 2.5.12 Switch **Evac/Auto/Fill** toggle (#10) to the middle position (**AUTO ON**).
- 2.5.13 **OPEN V3** and **V4**.
- 2.5.14 Proceed to the Post Cleaning Procedure (Steps 2.6.03-2.6.10).

2.6 Post Cleaning Procedure (Shutdown)

- 2.6.1 Turn off the ovens and open the oven doors to begin cool down period.
- 2.6.2 Check that all canister sampling valves and oven manifold valves are closed.
- 2.6.3 **CLOSE V1** (vacuum pump valve) and turn off the vacuum pump.

CAUTION: *SAFETY GLASSES & GLOVES ARE REQUIRED FOR STEP 2.6.4.*

- 2.6.4 Remove the dewar from the cold trap apparatus and dispose of the liquid nitrogen according to current safety guidelines.
- 2.6.5 Close the compressed nitrogen cylinder valve as well as the nitrogen regulator needle valve.
- 2.6.6 Remove canisters from the ovens.
- 2.6.7 Enter the appropriate information (i.e. contamination check number) into the Canister Logbook.

- 2.6.8 Turn Aadco purifier off only if the cleaning system will not be used for at least two days.
- 2.6.9 One canister per cleaning batch must be documented and analyzed by the gas chromatographic method for which they are used. Furthermore, the contamination check results for each batch must not exceed the established criteria listed in Table 1.

3. MANUAL PROCEDURE FOR CLEANING CANISTERS

SUMMARY OF STANDBY CONDITIONS

Valves: 1, 2, 5, 7	: CLOSED
Valves 3, 4, 6	: OPEN
Oven Manifold Valves	: CLOSED
Liquid N ₂ Valve	: CLOSED
Aadco Purifier	: ON or OFF
Pump	: OFF
Pressure Gauge	: 760 torr (atmospheric pressure)

3.1 Canister Cleaning Procedures

Please refer to **Figures 1-1** and **1-2** to interpret all component symbols and structures.

- 3.1.1 Turn the Aadco Purifier “**ON**” and open the liquid nitrogen headspace valve.
- 3.1.2 Make sure that the “Nitrogen” plug, “Pressurizing” plug, “Vacuum” plug and “Vent” plug are in place.
- 3.1.3 **CLOSE V3** (nitrogen inlet) and **V4** (vacuum inlet).
- 3.1.4 Set the mass flow controller (MFC) to 12 for Tylan RO-32 or to 60% for the Porter Instrument.
- 3.1.5 Position the 1600 ml dewar underneath the glass vacuum cold trap such that the glass finger is inside the dewar but not touching the inner surface of the dewar.
- 3.1.6 Secure all supports for the dewar-trap apparatus.

CAUTION: *SAFETY GLASSES & GLOVES ARE REQUIRED FOR STEP 3.1.7.*

- 3.1.7 **SLOWLY** fill a second dewar with liquid nitrogen, then **CAREFULLY** transfer the liquid nitrogen to the secured dewar. Level of liquid nitrogen should be about 1” from the top the dewar.
- 3.1.8 Ensure that **V2** (vacuum trap purge valve) is closed.

- 3.1.9 Make sure that **V1** (vacuum pump inlet) is closed, turn on the vacuum pump.
- 3.1.10 Manually **OPEN V1**.
- 3.1.11 Vent all eight pre-selected canisters in the safety fume hood before attaching them to the manifolds.
- 3.1.12 Log all canisters into the cleaning log book according to the oven manifold number (labeled in the oven) and canister number.
- 3.1.13 Connect as many as eight 6-liter canisters to the oven manifolds with the canister sampling valves closed. Place canisters in the back of the oven first. Tighten each manifold connection to the canister using a “back up wrench” (size 9/16).
- 3.1.14 Open the canister sampling valves.
- 3.1.15 **OPEN** all oven manifold valves.
- 3.1.16 **OPEN V4** (vacuum inlet).
- 3.1.17 When the pressure gauge of the canister cleaning system drops below **20 torr or < 1.0 psig**, **OPEN V5** (to vacuum gauge) and ensure that the system is able to evacuate to **< 1.0 torr**.
- 3.1.18 **CLOSE V4** and **V5**.
- 3.1.19 Turn on the ovens, which are preset at 55⁰C. Close and latch oven doors.
- 3.1.20 **OPEN V4** (vacuum inlet) to allow the system to evacuate. At this point, the needle for pressure gauge of the canister cleaning system should be decreasing to the vacuum range.
- 3.1.21 If there are no leaks attributed to the canister-oven manifold connections or the canister itself, the system will reach **<1.0 torr**.

THE CANISTER CLEANING CYCLE (Steps 3.1.22-3.1.23)

The canisters are filled with humidified nitrogen and then equilibrated for at least 7 minutes. A flow rate of 100 torr/25 seconds for 8 canisters is equivalent to a flow rate of 15 liters/min.

- 3.1.22 **Pressurizing canisters with humidified nitrogen:**
 - 3.1.22.1 Make sure that the “nitrogen” outlet plug, “Pressurize” outlet plug, “Vacuum” outlet plug and “Vent” outlet plug are in place.
 - 3.1.22.2 **CLOSE V4** and **V5** (to vacuum gauge.)

- 3.1.22.3 **SLOWLY OPEN V3** (nitrogen inlet) and fill the canisters to 20 psi as indicated on the pressure gauge of the canister cleaning system.
- 3.1.22.4 **CLOSE V3.**
- 3.1.22.5 Equilibrate canisters for 7 minutes,
- 3.1.23 **Evacuating the canisters:**
- 3.1.23.1 Remove all plugs except the “ Vacuum” outlet plug.
- 3.1.23.2 **SLOWLY OPEN V4.**
- 3.1.23.3 When the pressure gauge of the canister cleaning system reads **<20 torr, OPEN V5.**
- 3.1.23.4 As the pressure drops **below 1 torr**, go back to Step 3.1.22.
- 3.1.23.5 Complete steps 3.1.22-3.1.23 four more times (5 total) ensuring that there is ample supply of liquid nitrogen in the cold trap dewar at all times.
- 3.1.23.6 If the canister(s) require **PRESSURIZATION**, proceed to Section 3.2. If the canister(s) must remain **EVACUATED**, proceed to Section 3.3.

3.2 To Pressurize a Cleaned Canister

- 3.2.1 Be sure that all canisters, except those to be pressurized, are closed at the canister sampling valves and the oven manifold valves.
- 3.2.2 **CLOSE V4** (vacuum inlet).
- 3.2.3 **With** the “ Pressurize” outlet plug in place, **SLOWLY** (100 torr/25 sec) **OPEN V3** (nitrogen inlet) and fill the pre-selected canister(s) to the required final pressure reading.
- 3.2.4 **CLOSE** the canister sampling valves and the oven manifold valves.
- 3.2.5 **CLOSE V3.**
- 3.2.6 Proceed to the Manual Post Cleaning Procedure (Section 3.4).

3.3 To Maintain Evacuated Status of Cleaned Canisters

- 3.3.1 After the cleaning steps 3.1.22-.3.1.23, all canisters should be at a vacuum no less than **–30 inches Hg.**
- 3.3.2 Ensure that all canisters that must remain evacuated close all canister sampling valves and manifold valves.

3.3.3 Proceed to the Manual Post Cleaning Procedure (Section 3.4)

3.4 Manual Post Cleaning Procedure (Shutdown)

- 3.4.1 Turn off the ovens and open the oven doors to allow them to cool
 - 3.4.2 Check to see if all canister sampling valves and oven manifold valves are closed.
 - 3.4.3 Make sure that “Nitrogen” plug, “Pressurize” plug, “Vacuum” plug, and “Vent” plug are in place.
 - 3.4.4 If the system pressure is >> **760 torr**, vent the system by opening **V7** (vent valve) to allow the pressure to drop to approximately 800 torr. Close **V7** and proceed to step 3.4.7.
 - 3.4.5 If the system pressure is between **760 torr** and **800 torr**, go to step 3.4.7
 - 3.4.6 If the system pressure is <<**760 torr**, use procedure listed in steps 3.4.6.1 through 3.4.6.4.
 - 3.4.6.1 **CLOSE V5** (to vacuum gauge).
 - 3.4.6.2 **CLOSE V4** (vacuum inlet).
 - 3.4.6.3 **SLOWLY, OPEN V3** (zero air inlet) to pressurize the system to **approximately 800 torr**.
 - 3.4.6.4 When **800 torr** is reached, **CLOSE V3** and proceed to step 3.4.7.
 - 3.4.7 **CLOSE V1** (vacuum pump valve) and turn off the vacuum pump.
- CAUTION:** *SAFETY GLASSES & GLOVES ARE REQUIRED FOR STEP 3.4.8.*
- 3.4.8 Remove the dewar from the cold trap apparatus and dispose of the liquid nitrogen according to current safety guidelines.
 - 3.4.9 Shut off the nitrogen supply by closing the nitrogen cylinder valve as well as the nitrogen regulator needle valve.
 - 3.4.10 Remove the canisters from the ovens.
 - 3.4.11 Enter the appropriate information (i.e. contamination check number) into the Canister Logbook.
 - 3.4.12 Turn the Aadco purifier off only if the cleaning system will not be used for at least two days.
 - 3.4.12.4 One canister per cleaning batch must be documented and analyzed by the gas chromatographic method for which they are used. Furthermore, the

contamination check results for each batch must not exceed the established criteria listed in Table 1.

SUMMARY OF STANDBY CONDITIONS

Valves: 1, 2, 5, 7	: CLOSED
Valves 3, 4, 6	: OPEN
Oven Manifold Valves	: CLOSED
Liquid N ₂ Valve	: CLOSED
Aadco Purifier	: ON or OFF
Pump	: OFF
Pressure Gauge	: 760 torr (atmospheric pressure)

3.5 Canister Evacuation Only (no cleaning)

*Please note that procedures in section 2.4 are applicable if the canister conditioner controller will be used or automation is desired.

- 3.5.1 Make sure that only the "Vacuum" outlet plug and "Vent" outlet plug are in place.
- 3.5.2 Close **V2** (vacuum trap purge valve), **V3** (nitrogen inlet), **V4** (vacuum inlet), canister sampling valves, and oven manifold valves. The ovens do not need to be on for this procedure.
- 3.5.3 Connect as many as eight 6-liter canisters to the oven manifolds. Place canisters in the back of the oven first, and tighten them down employing a wrench (size 9/16").
- 3.5.4 Place the 1600 ml dewar underneath the glass vacuum cold trap such that the glass finger is inside but not touching the inner surface of dewar.
- 3.5.5 Secure all supports for the dewar-trap apparatus.

CAUTION: SAFETY GLASSES & GLOVES ARE REQUIRED FOR STEP 3.5.6.

- 3.5.6 **SLOWLY** transfer liquid nitrogen via a second dewar into the secured cold trap dewar containing the glass finger and fill to about 1 inch from the top of the dewar.
- 3.5.7 While **V1** (vacuum pump inlet) is **CLOSED**, turn on the vacuum pump.
- 3.5.8 **OPEN V1** manually.
- 3.5.9 **OPEN** the oven manifold valves and **V4** (vacuum inlet).
- 3.5.10 When the pressure gauge of the canister cleaning system drops below **20 torr or < 1.0 psig**, **OPEN V5** (to vacuum gauge) and ensure that the system can evacuate to **< 1.0 torr**.

- 3.5.11 **CLOSE V4 and V5.**
- 3.5.12 If the canisters are above **15 psig**, **CLOSE V6** (to pressure gauge). **OPEN** the canister sampling valves, then **QUICKLY OPEN V7** (vent valve) to allow canisters to reach just above 760 torr.
- 3.5.13 **CLOSE V7**, then begin evacuating by **opening V4**.
- 3.5.14 When the pressure gauge of the canister cleaning system drops below **1 torr**, **CLOSE** the canister sampling valves and the oven manifold valves.
- 3.5.15 **CLOSE V4.**
- 3.5.16 Proceed to Manual Post Cleaning Procedure, Steps 3.4.3-3.4.12.
- 3.6 Pressuring Previously Cleaned Canisters (No Cleaning)
- 3.6.1 Turn the Aadco purifier "**ON**".
- 3.6.2 Make sure that the "nitrogen" outlet plug, "Pressurize" outlet plug, "Vacuum" outlet plug, and "Vent" outlet plug are in place.
- 3.6.3 Close **V2** (vacuum trap purge valve), **V3** (nitrogen inlet), **V4** (vacuum inlet), canister sampling valves, and oven manifold valves. The ovens do not need to be on for this procedure.
- 3.6.4 Set the mass flow controller (MFC) to 12 for the Tylan RO-32 or to 60% for the Porter Instrument.
- 3.6.5 Proceed with Canister Evacuation Steps 4.1.4-4.1.12.
- 3.6.6 Pressurize the pre-selected canister(s) by opening the canister sampling valves and oven manifold valves. The system and canister(s) should initially be <1.0 torr.
- 3.6.7 **OPEN V3** (nitrogen inlet), and allow the canister(s) to pressurize to the required level indicated at the pressure gauge of the canister cleaning system.
- 3.6.8 **CLOSE** the canister sampling valves and the oven manifold valves.
- 3.6.9 **CLOSE V3.**
- 3.6.10 Vent excess pressure in the system by **OPENING V7** (vent valve), then **CLOSE V7** when the system has decreased to about 800 torr.
- 3.6.11 Proceed to Manual Post Cleaning Procedure, Steps 3.4.7-3.4.13.

Table 1: CRITERIA LEVEL FOR CLEAN CANISTERS

Contaminant	Toxic Criteria Level (ppb)
Benzene	< 0.50
Carbon tetrachloride	< 0.02
Chloroform	< 0.02
Ethylene dibromide	< 0.01
Ethylene dichloride	< 0.20
Dichloromethane	< 1.0
Perchloroethylene	< 0.07
Trichloroethane	< 0.02
1, 1, 1- Trichloroethane	< 0.10
Contaminant	NMOC Criteria Level (ppb-C)
Speciated-per component	1.0
Total PDFID	30.0

FIGURE 1-1 . Pressurizing Canisters with Humidified Zero Air

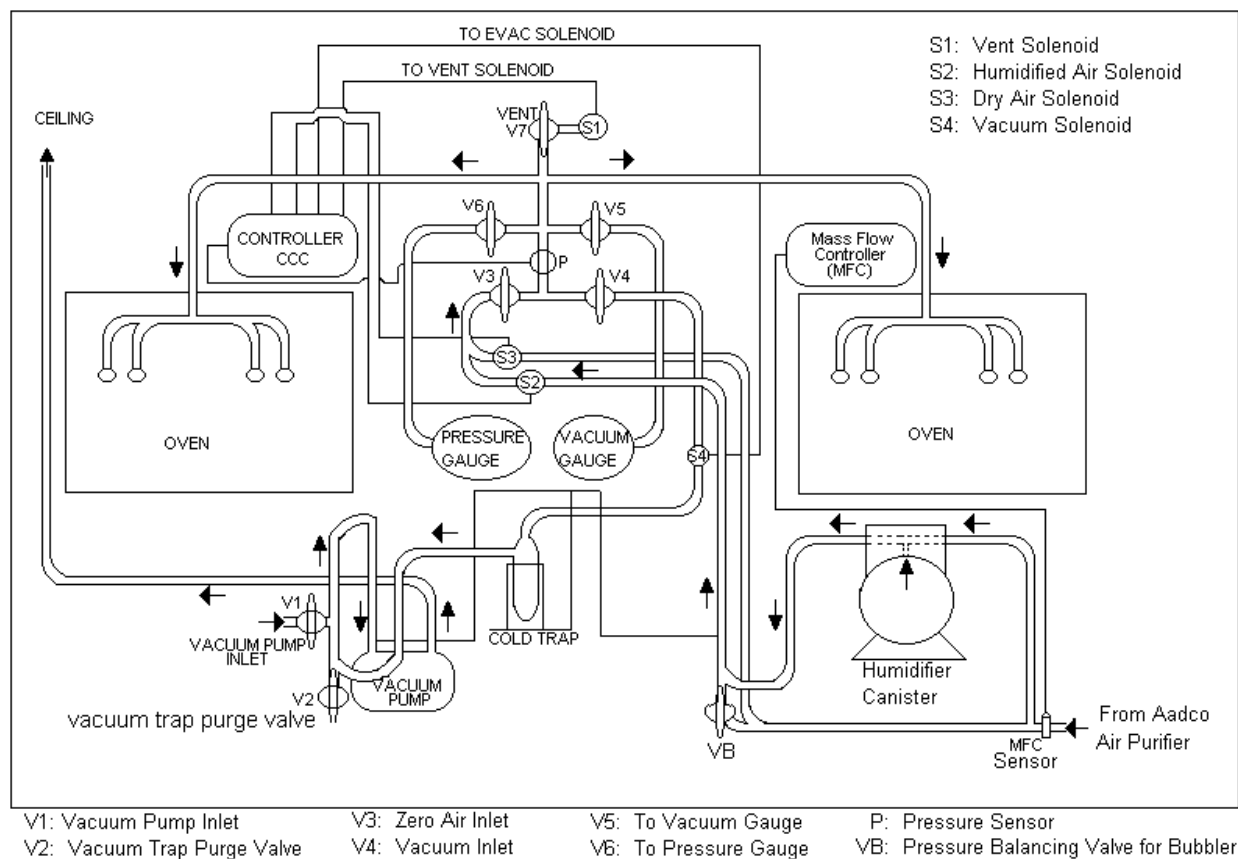


FIGURE 1-2. Canister Evacuation

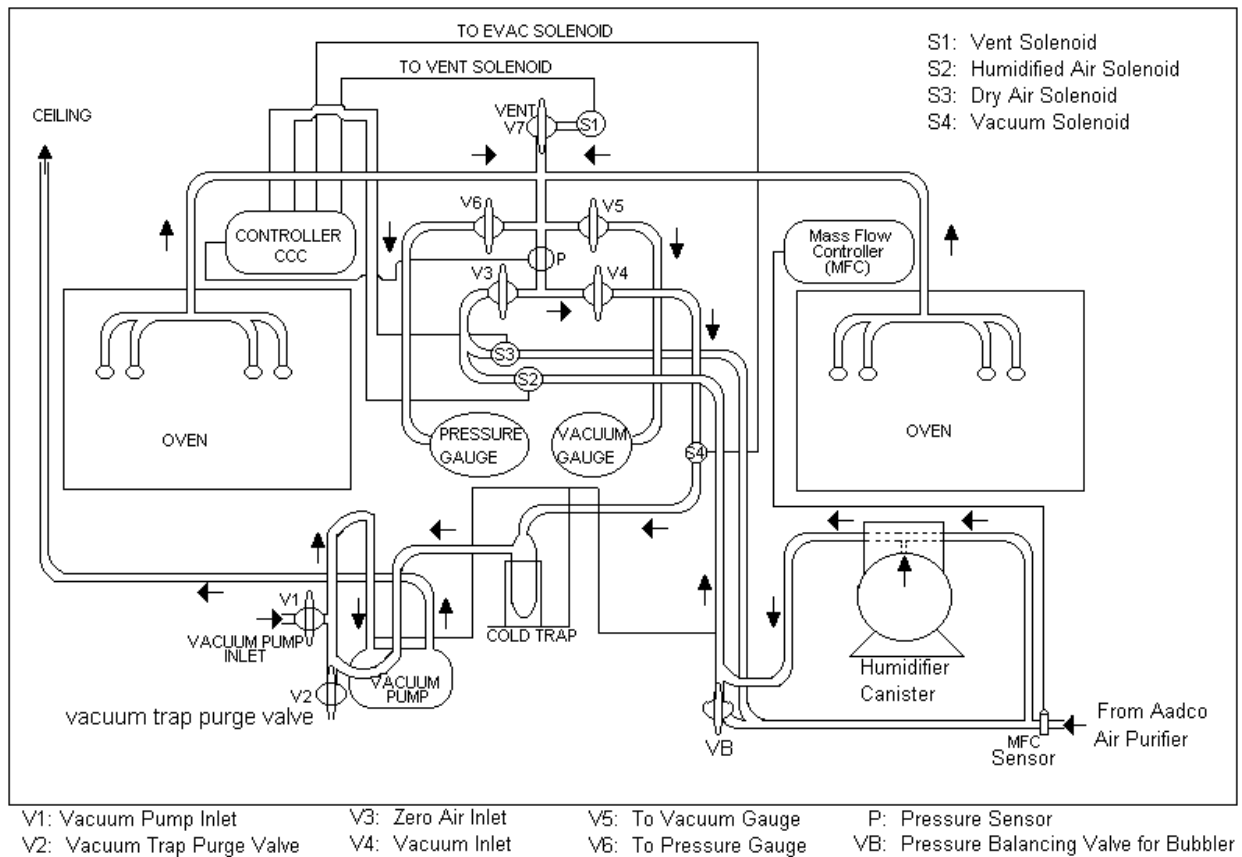


FIGURE 2. Diagram of Canister Conditioner Controller

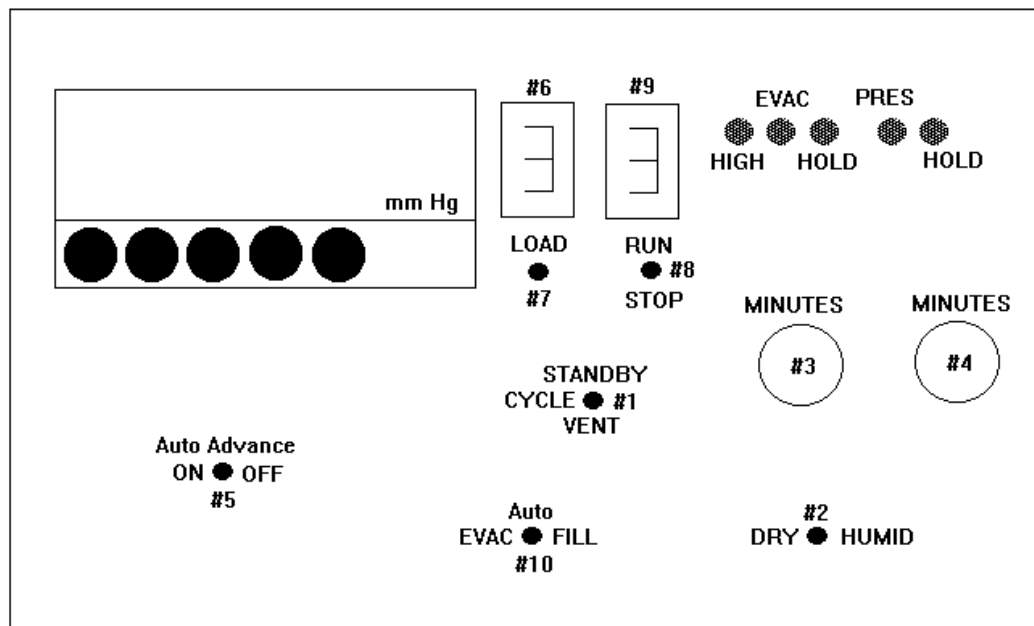


FIGURE 3. Canister Conditioner Controller Input/Output Schematic

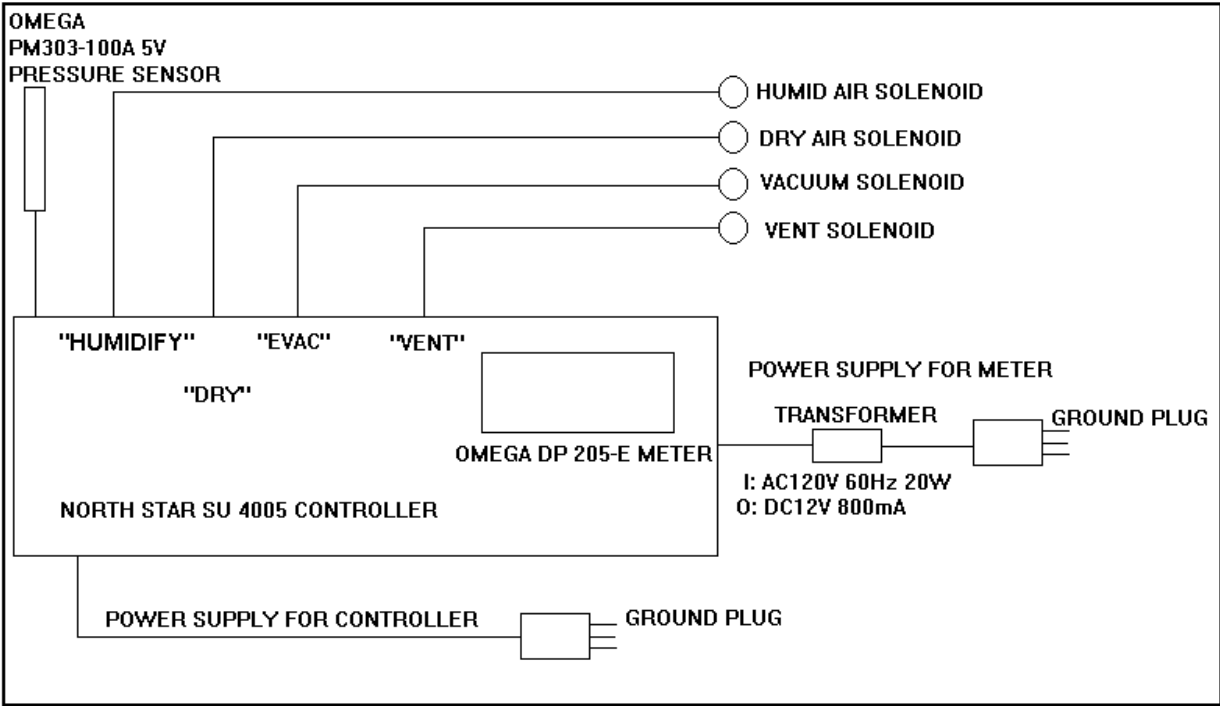
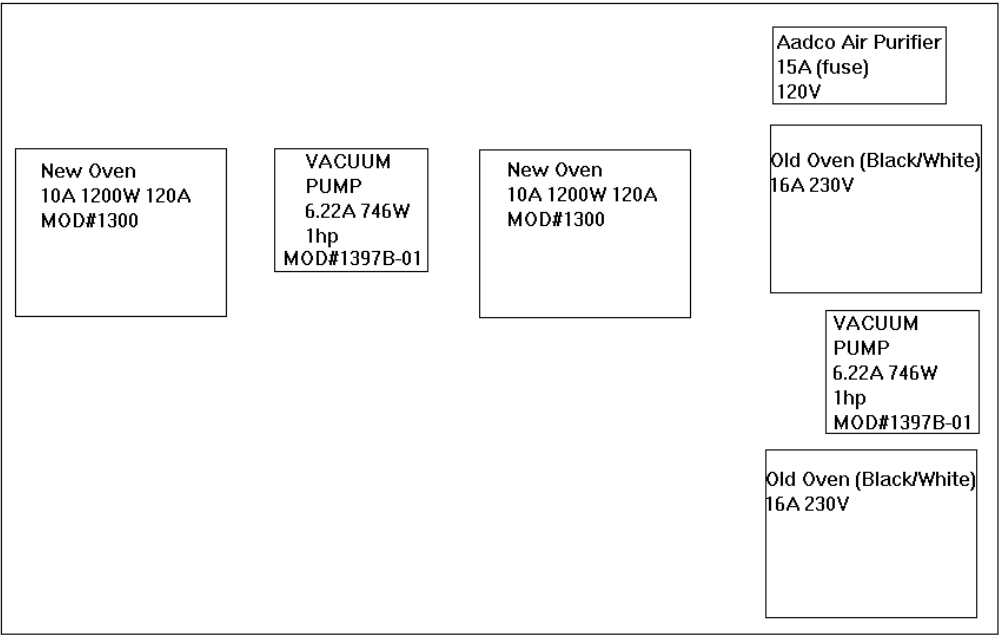


FIGURE 4. Canister Cleaning Room - Electrical Diagram



APPENDIX

**CANISTER CONDITIONER CONTROLLER OPERATING
INSTRUCTIONS**

BY
NORTH STAR INSTRUMENTS, CAMARILLO, CALIFORNIA.

APRIL 1994

PART A

DESCRIPTION AND OPERATION

Upon power up, or after a power interruption lasting over a second, the Cycle counter will read zero, and if a program was in progress, it will be stopped with all the valves closed and stage indicators off. Momentary power interruption “glitches” may cause erroneous operation and false triggering if proper spike protection is not provided for both AC power cords.

The Sequence will not run if a **ZERO** appears in the display and all valves are closed.

The Cycle Display will decrease by one at the completion of each pressurized hold period. When the counter reaches zero, the canisters are evacuated, the evac valve will remain open, and the sequence stops.

Press Stop to close the evac valve. If the evacuation valve is still open from the previous cycling, the sequence may be run again by simply pressing the **LOAD** switch.

When Auto Advance is **ON**, the appropriate valve will be open until the respective setpoint is reached and remains for 12 seconds. With the control off, the setpoint controlled advance delay feature is bypassed.

If the number of cycles and hold times are not changed, the only steps to take to run another set is to press the counter load and then the start switches.

The number of cycles may be changed and loaded anytime during a sequence without stopping or affecting the sequence.

Starting the sequence while a run is already in progress will cause a restart at the evacuation period, stopping the other stages. Press **STOP** at any time to cancel a sequence.

Manual valve activation will stop a sequence, and it cannot be restarted while any valve is manually energized.

In addition, the counter display will go blank. When the VENT valve is activated, the decimal point LED in the counter display window will illuminate. All other LEDs will be out.

CONTROLLER OPERATION OVERVIEW

1. Configure the Panel Meter. Program the setpoints.
2. Program the number of cycles by thumb wheel selector, then press the Load Switch. The number will appear in the display.
3. Set the Auto Advance Control to **ON**.
4. Set desired Hold Times.

5. Select Dry or Humid for the Pressurize mode. It may be changed anytime during a sequence.
6. Valve selectors on **AUTO**.
7. Press Start.

For testing and performance checks, load the desired number of cycles, set the Pressure/Vacuum Control switch to OFF, set both Timers to zero, then press Start. The four step sequence will be completed in about 45 seconds, and will recycle for as many times as was programmed.

PANEL METER SETUP

The DIP Switches and connections have been made. Only front panel data entries are required.

Following the procedure in the Operator's Manual to program the Parameters listed below. To configure the Panel Meter:

1. Select 10 V for the 0-10 V DC input range.
2. Enter the Decimal Point to far right.
3. To scale "input one" using the supplied sensor, enter $.5 \times 1000 = 500$ and "input two" should be $5.5 \times 1000 = 5500$. "Read one" should be 0000, and "Read two" should be 5170. The above scaling is based upon 100 PSI= 5170 torr.
4. Use the filtered input signal unless problems with it occur.
5. Set point one is for vacuum advance control. Select "active below" Set point and the "unlatched" mode.
6. Set point two controls the pressure advance. Select "active above" Set point and the "unlatched" mode.
7. Output configuration should be: enabled, regular, voltage.
8. Analog Output scale and offset: The analog output "Read one" should be 0 V=0000 with "out one" also showing 0000 V. "Read two" can either be 5170 or 517 (refer to section regarding vacuum fine adjusted Set point). "Out two" should be 10.00 V.

The Analog Output Signal is available on the back panel for connection to a DVM if necessary to view the Panel Meter performance.

SET POINTS

The Panel Meter relays are activated when the programmed digital display setpoint of the meter is reached. Therefore, at the evacuation control level, an intermediate signal must be provided between 0000 and 0001 when millitorr setpoint control is needed. To accomplish this, the analog output of the Panel Meter is conditioned by the Main Controller for continuous millitorr setpoint adjustment, using a trim pot accessible through the right side of the enclosure. An indicator LED labeled "HIGH" will be ON whenever the pressure is higher than the vacuum Fine Setpoint.

The Vacuum setpoint control (output 1) from the Panel Meter and the Fine Setpoint work in parallel. The lowest of your two vacuum setpoint methods will control the auto-evacuation delay function. Because of this, it is important that the Panel Meter setpoint be programmed higher than is needed when using the fine adjustment capability.

Although the Fine setpoint is preadjusted for about 500 millitorr (based on 10 V =5170 torr), it may be changed as follows:

On the Panel Meter, select 2 torr (or higher) for setpoint one, then while observing the high” LED, vary the fine adjust so that the LED just goes out when your desired vacuum (less than one torr) is reached. As long as the “High” (fine Setpoint) LED is on , the evacuation valve will remain open when at the evacuation period. The Fine Setpoint LED status has no effect upon the other three stages. If you do not desire to use the fine adjustment, set it higher than necessary, then program the Panel Meter for the required torr setpoint, which would then be lower than the fine adjustment setting. The range of the Fine Adjustment is from zero to about 40 torr when the Panel Meter analog out is programmed for 10 V=5170 torr. If the analog out is programmed for 10 V= 517 torr, an amplification of 10 will then resale the fine adjust so that the maximum range will be 4 torr. As a result, there will be 10 times more resolution in which to place a setpoint.

The Pressure setpoint has no fine adjustment. Only the Panel Meter requires programming (setpoint two). Example; 2600 torr.

NOTE 1: It is important that both the pressure sensor and the controller be maintained at room temperature. Variations in temperature more than a few degrees could result in a shift in the evacuation setpoint.

NOTE 2: Do not place the controller near sources of high current AC as this may cause erratic operation. Separation of at least a foot is recommended.

As with any electronic device, avoid moisture, shock, heat, and flammable vapors.

If operational difficulties should arise, contact NSI for assistance. Do not attempt to troubleshoot the controller, or remove components. If repairs are required, the entire unit must be returned for thorough testing. The only exception is if a known problem develops with pressure sensor, in which case it may be dealt with independently.

WARRANTY Canister Conditioner Controller SN 4005

North Star Instruments warrants the Canister Conditioner Controller to be free from defects in material and workmanship for a period of 90 days, the Panel Meter for one year, and the sensor for three years from date of shipment. This warranty does not cover damage resulting from accident, misuse, neglect, modification, tampering, improper operation or shipping damage. Claims of shipping damage must be made to the carrier. This warranty shall extend only to the original purchaser of the controller for use by the purchaser, and for the specific utilization for which it was designed.

North Star Instruments shall in no event be liable for any incidental or consequential injury or damages of any kind; the sole obligation of the manufacturer is limited to repair

or replacement of defective products. Some states do not allow exclusion or limitation of incidental or consequential damages, so the above limitations or exclusions may not apply to you.

For service under this warranty, return the controller shipping prepaid to:

North Star Instruments
44 Calvados Dr.
Camarillo, CA 93012

For technical assistance, questions, warranty service, subcomponent replacement or repairs after warranty, call NSI at (805) 484-7892.

PART B

MAJOR COMPONENTS OF THE CANISTER CONDITIONER CONTROLLER

Vendor #1: Omega Engineering Inc.
P.O. Box 4047
Stanford, CT 06907

STOCK ITEM#	DESCRIPTION
PX303-100A5V	Pressure Transducer 0-100 PSIA (0.5 – 5.5 V Output)
PS-4G	Snubber
DP25-E-AR	Process Meter 0-10V 12V Excitation

PART C

Procedures for re-starting Canister Conditioner Controller after Power Failure

1. Start the canister cleaning system by following the automated canister cleaning SOP MLD020 (Revision 3.2)

procedures (Section 2).

2. When the digital pressure display on the controller reaches 100 torr, open V5
3. When the vacuum gauge on the front panel of the cleaning system has dropped below 1 torr, close V5.
4. Press "Tare" on the digital gauge. The system should be pressurized within two minutes as evident by the increasing pressure on the digital display.